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ERDEC-CR-238

TEMPERATURE MEASUREMENTS TO CHARACTERIZE DISPERSION WITHIN PRESSURE SWING ADSORPTION (PSA) BEDS

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13. ABSTRACT *(Maximum 200 words)*

PSA experiments using a two-bed, laboratory-scale (100 standard liters per minute [SLPM]) system equipped with in-bed temperature and vapor-phase concentration probes, were performed using trichlorofluoromethane (R-11) as the feed contaminant and air as the carrier. In-bed, vapor-phase concentrations at each axial position were measured and correlated to the magnitude of the temperature swings during a cycle. The PSA experiments were then conducted using a full-scale (5,660 SLPM) system with difluorochloromethane (R-22) and 13X molecular sieve. Fourteen temperature probes were placed within a bed at different radial and axial positions. Using the temperature-concentration correlation, it was possible to construct a visual representation of the dispersion characteristics of the larger beds.

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PREFACE

The work described in this report was authorized under Contract No. DAAA15-93-C-0070. This work was started September 1994 and completed September 1995.

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1. INTRODUCTION

The design and operation of adsorption-based separation and purification systems require a quantitative understanding of the important adsorption equilibria relationships. For many applications, particularly those where the desired separation is greater than 99%, one must also quantify the appropriate mass transfer resistances and axial dispersion. Costa and Rodrigues[1] demonstrated the significance of axial dispersion in their analysis of a liquid-phase adsorption process. Mahle and Friday[2] showed that axial dispersion contributes greatly to the spreading of the adsorption wave for vapor-phase adsorption systems, particularly for more strongly adsorbed vapors. Both works developed material balances for a cylindrical adsorber. Mass transfer rates were determined using engineering calculations and batch uptake experiments. All bed-scale effects (e.g., deviations from plug flow) are lumped into the axial dispersion term. This work demonstrates experimentally the effect of bed diameter on dispersion and the impact of axial dispersion on system performance.

2. EXPERIMENTS

Two systems are used to generate the data described in this work. The low flow system, hereafter called the lab-scale system, operates at feed flow rates between 75 and 225 standard liters per minute (SLPM), while the high flow system, designated as the full-scale system, is capable of feed flow rates up to 6300 SLPM. Mass flow controllers are used on the product and feed streams. A regulating valve is used on the purge to set the system pressure. A solenoid valve is used downstream of the purge flow regulating valve to perform the 4-step cycle. The lab-scale system uses four 3-way valves to direct flows. The full-scale system uses two 4-way slide valves.

Both systems use two-beds equipped with multiple in-bed vapor-phase concentration and temperature probes. For the lab-scale system, the temperature and concentration probes are placed in 5-cm increments from the inlet of the bed to the outlet. The probes are inserted through the wall of the beds and sample gas from the center of the bed. For the full-scale system, concentration probes sample vapor from the center of the bed, while the temperature probes are positioned according to Table 2. The time constant for all temperature probes is about 1 second. Both the lab-scale and full-scale systems use a novel vapor-phase sampling system which allows one to obtain a complete (at each designated probe position) bed profile at a selected time during a cycle.[3]

3. RESULTS

Experiments were performed using lab-scale and full-scale PSA systems. Results from the lab-scale system were used to establish a correlation between the magnitude of the temperature swings during a cycle and the concentration of adsorbed contaminant. The operating conditions used for each experiment are given in Table 1.

Table 1. Conditions used for the lab-scale and full-scale PSA experiments

Parameter	Lab-scale Experiment	Full-scale Experiments
Adsorbent	BPL Activated Carbon	13X molecular sieve
Adsorbate	R-11	R-22
Bed Diameter	3.81 cm	33 cm and 23.6 cm
Bed Depth	24 cm	24 cm
Feed Flow Rate	100 SLPM	5660 SLPM
Product Flow Rate	50 SLPM	2830 SLPM
Purge Flow Rate	50 SLPM	2830 SLPM
Feed Pressure	48 psig	60 psig
Feed Concentration	4.0 mg/lit	4.0 mg/lit
Cycle Time	30 s	30 s

3.1 Lab-Scale Experiments.

PSA experiments using a two-bed, laboratory-scale with in-bed temperature and vapor-phase concentration measurements, were performed using trichlorofluoromethane (R-11) as the feed contaminant and air as the carrier. Prior to introducing the chemical into the system, temperature swings of more than 10 K were measured at each axial position within the bed during each cycle. It was determined by Brady, et al.,[4] in previous experiments conducted without contaminant chemical that air adsorbing and desorbing during the pressurization and blowdown steps was primarily responsible for the temperature swings.

Shown in Figure 1 are the results for the R-11/BPL carbon PSA experiment using the conditions given in Table 1. All results in Figure 1 represent measured data. The lines correspond to the difference between highest and lowest temperature measured during a cycle and the symbols correspond to the vapor-phase concentrations. All temperatures and concentrations are measured at the center of the bed. When R-11 is introduced into the feed, the magnitude of the temperature swings at each axial position begins to decrease as R-11 is detected at that position. As a result of adsorbed contaminant reducing the quantity of air adsorbed and desorbed during pressurization and blowdown, a correlation can be established between magnitude of the temperature swing at a given axial position and the vapor-phase concentration at the same axial position.

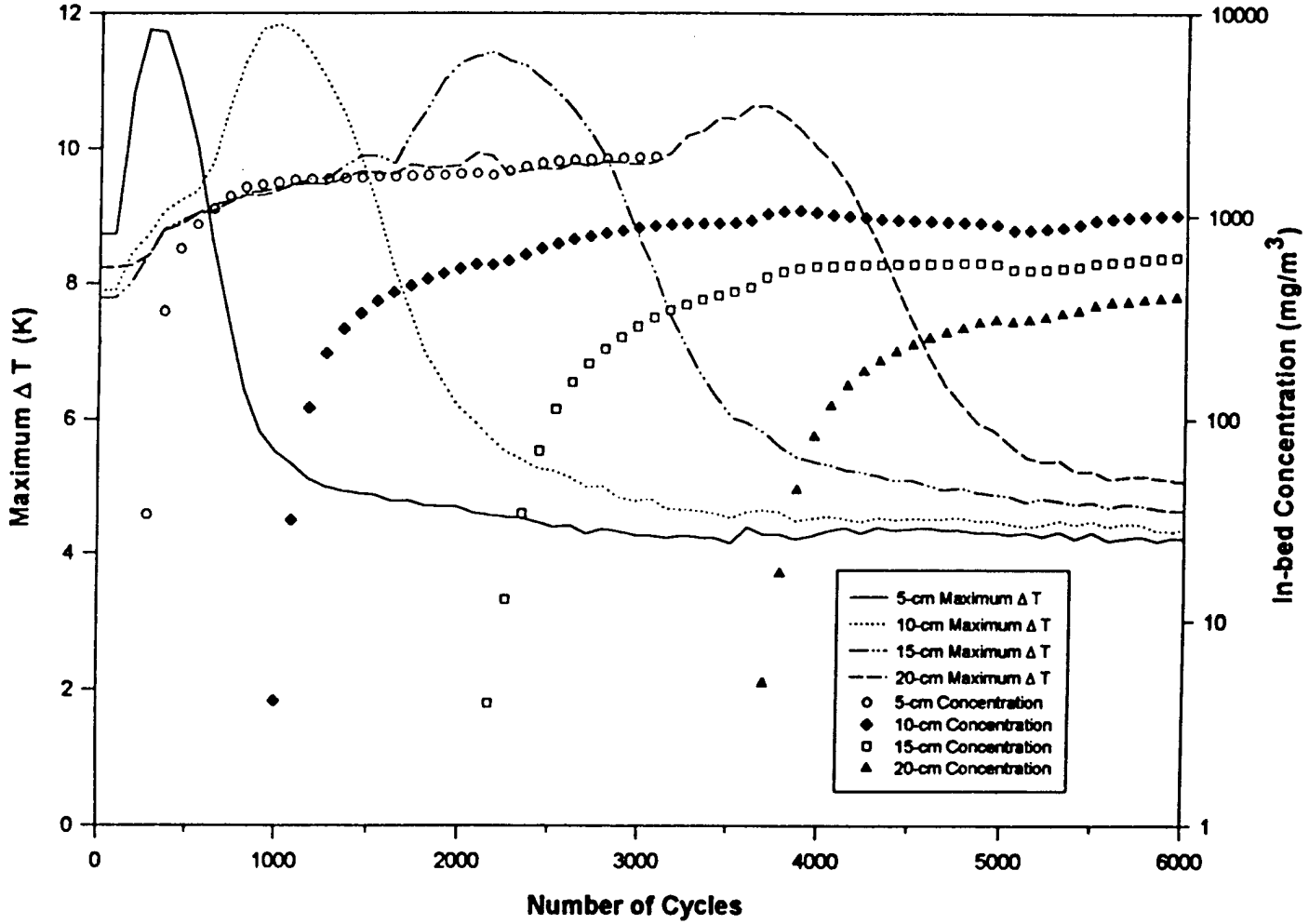

Figure 1. Results for R-11 on BPL Carbon. Maximum Δ T and In-bed Concentrations

Shown in Figure 2 are the measured temperatures for two cycles (1 minute) beginning after 90 cycles with chemical feed. Notice that all in-bed temperatures are below ambient and that the average magnitude of the temperature swings about 10 K. This behavior is discussed in detail by Mahle, et al.*

Shown in Figure 3 are temperature data for 2 cycles beginning at cycle 990. Referring back to Figure 1, the 5-cm position has broken through and almost reached its periodic-state value. Chemical has not been detected at the 10-cm position, however, it is less than 100 cycles from being detected. Note the increase in temperature during the feed step at the 10-cm position. This is probably caused by the heat of adsorption associated with adsorption front of R-11 which is in close proximity. At the end of the feed step, the temperature at the 10-cm position has increased about 4 K (from 292 K to 296 K). It is this behavior which results in the rise in the maximum Δ T shown in Figure 1 just prior to the first detectable concentration measurement.

3.2 Full-Scale Experiments.

The correlation established between the magnitude of the temperature swings during a cycle and the presence of adsorbed vapors was used to characterize the axial dispersion effects in larger beds. Experiments were performed using the full-scale conditions identified in Table 1. Run 43 used the larger diameter beds (33 cm) and Run 44 used the smaller diameter beds (23.6 cm).

Shown in Figures 4 and 5 are the concentration results for Run 43 and Run 44, respectively. There are several important observations from this data. Most important compare the relative positions of the 21-cm and product breakthrough curves. For Run 43 using the larger beds, R-22 is measured in the product approximately 100 minutes prior to the first measurable concentration at the 21-cm position. This demonstrates the level of dispersion. For the smaller diameter beds, Run 44, the product appears about 100 minutes after the 21-cm position.

Fourteen temperature probes were used within the bed. Table 2 identifies the axial and radial location of each of the 14 temperature probes. The radial position, r, is the distance from the center of the bed in cm. The axial position, z, is the distance from the feed inlet in cm.

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* Mahle, J.J., Friday D.K., and LeVan, M.D., Temperature Cycling and Subcooling in Pressure Swing Adsorption: Role of Weakly Adsorbed Carrier Gas in Purification Processes, U.S. Army Edgewood Research, Development and Engineering Center, Aberdeen Proving Ground, MD, unpublished data.

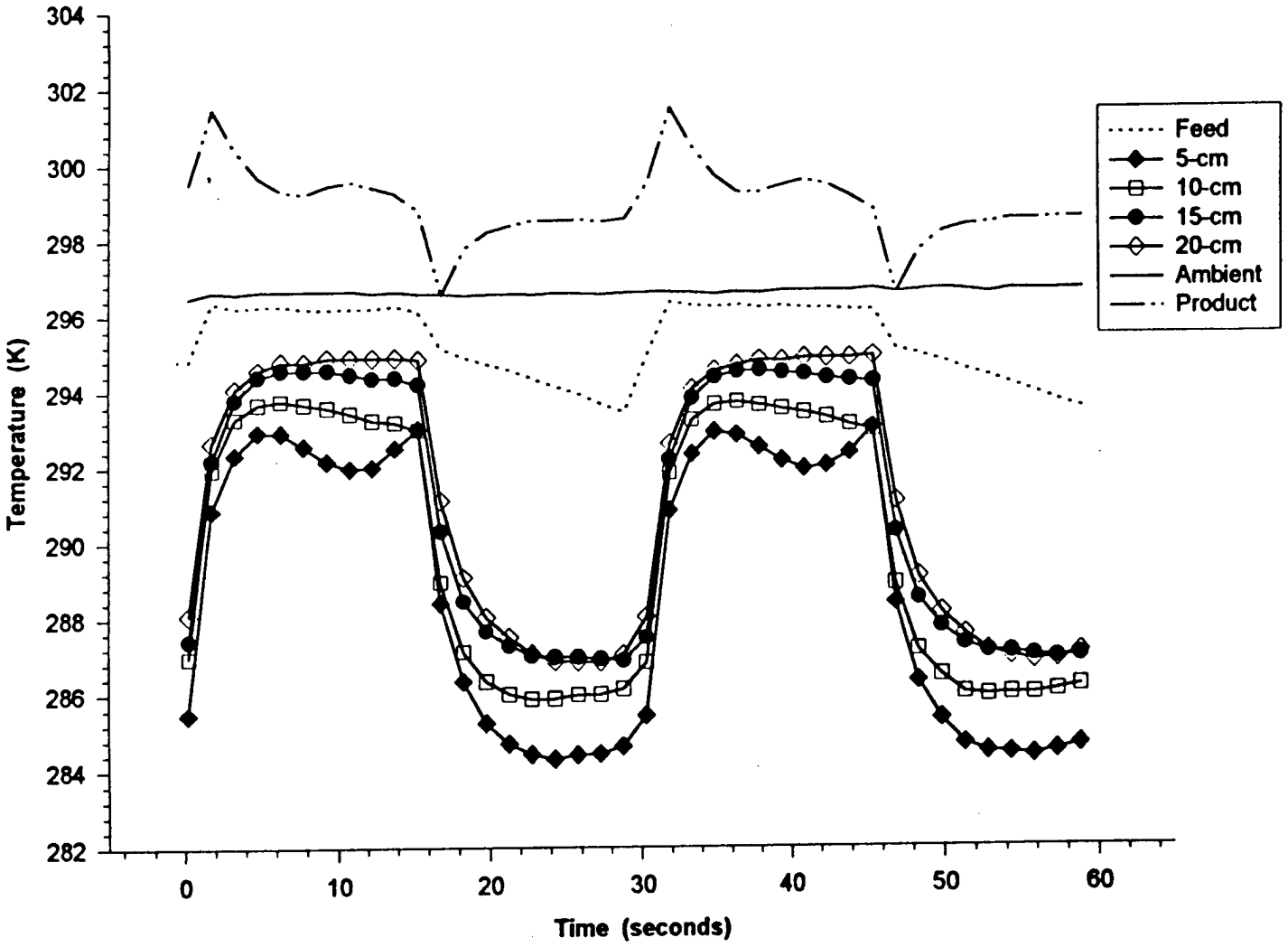

Figure 2. Lab-Scale Temperatures for 2 Cycles Starting at Cycle 90

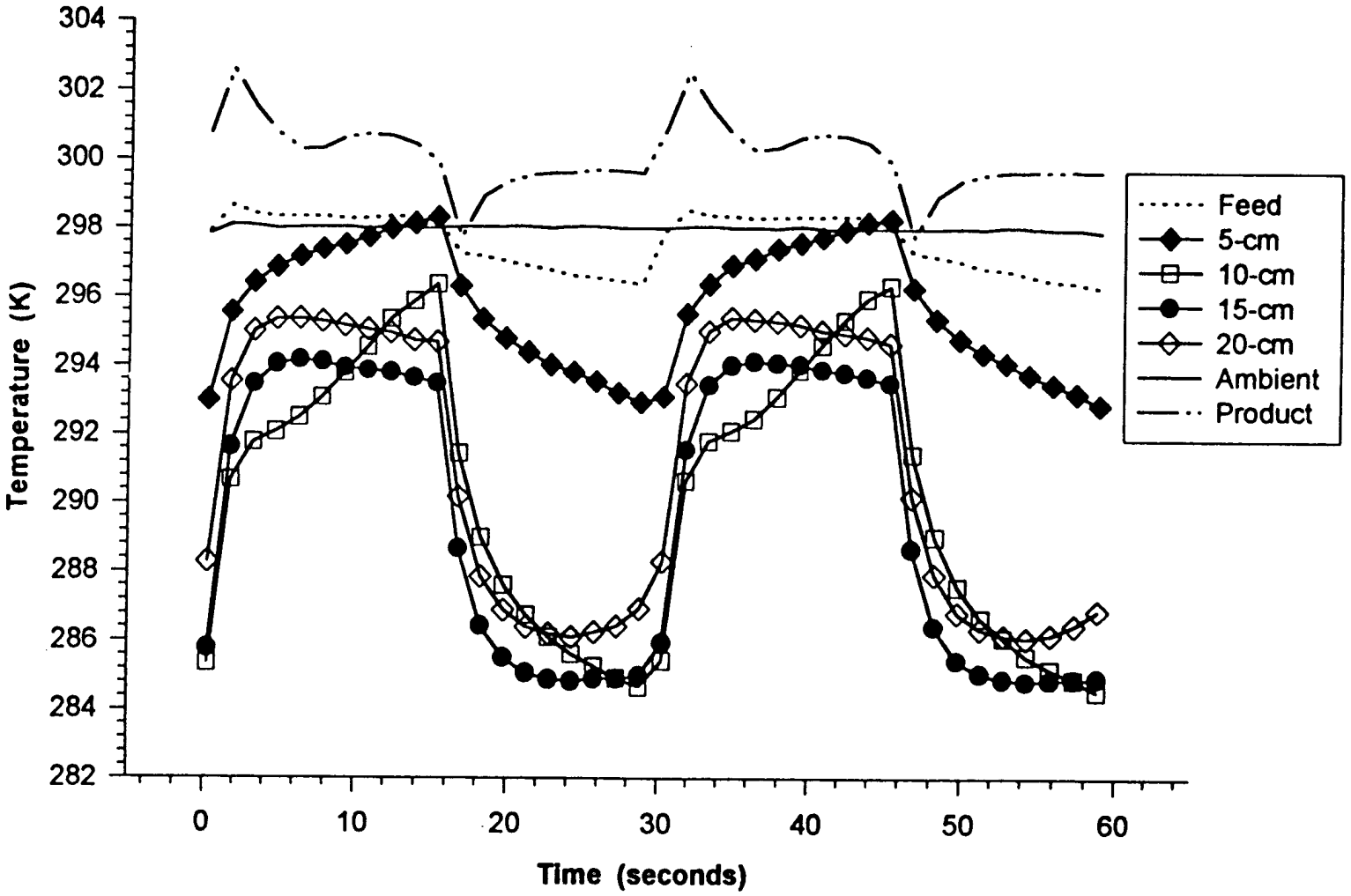

Figure 3. Lab-Scale Temperatures for 2 Cycles Starting at Cycle 990

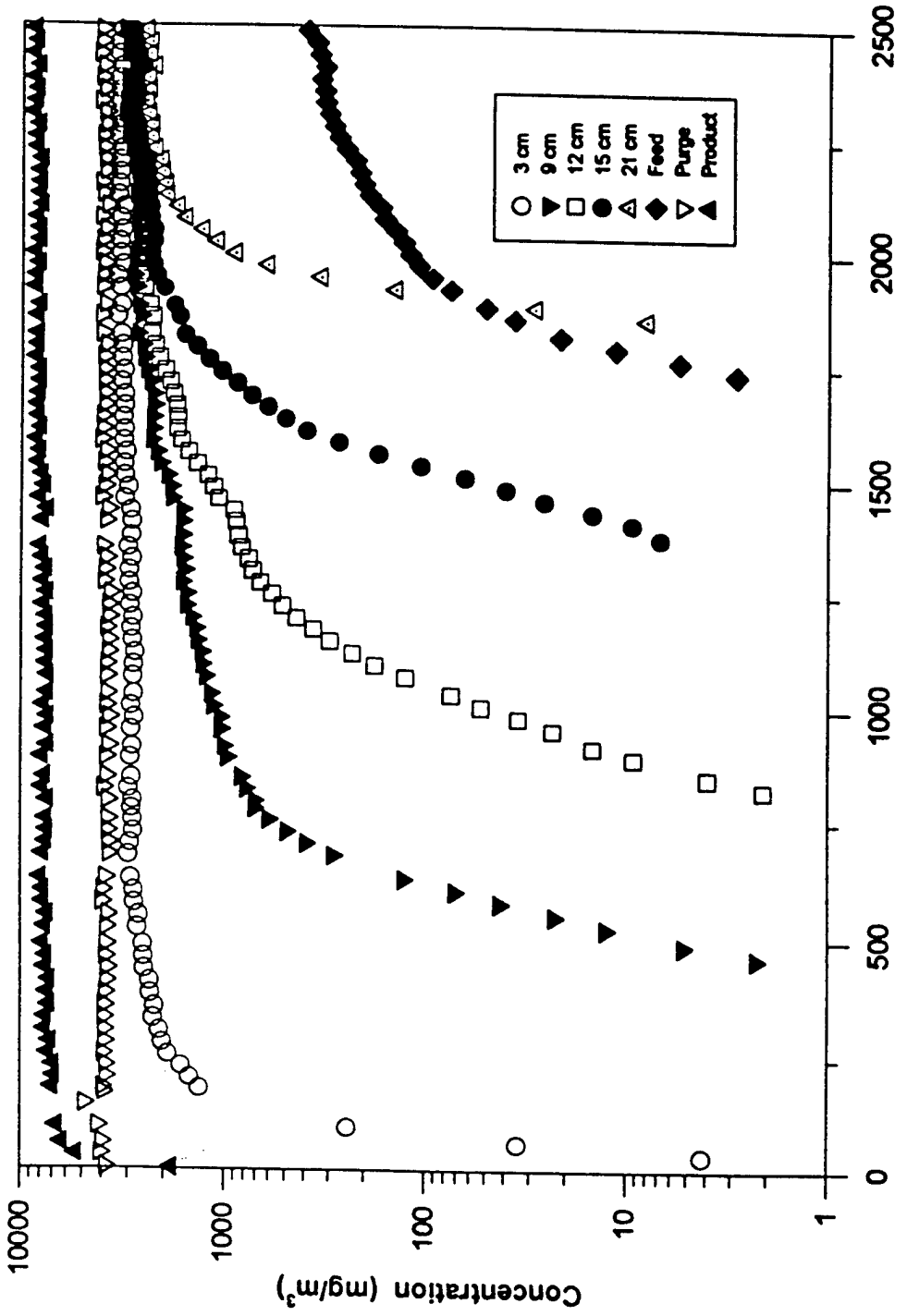

Figure 4. Run 43 Concentration Results (33-cm Diameter Beds)

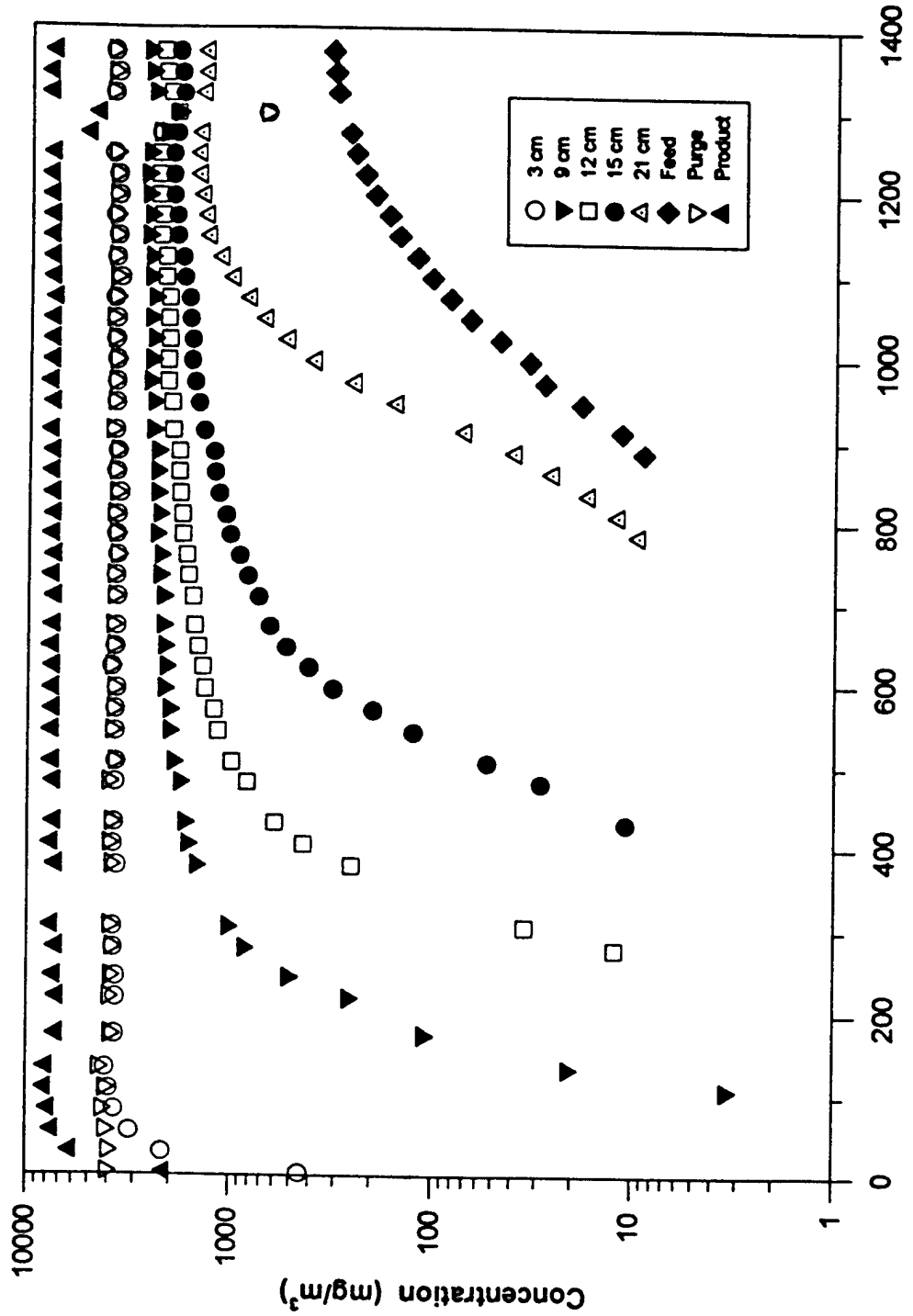

Figure 5. Run 44 Concentration Results (23.6-cm Diameter Beds)

Table 2. Positions of the Temperature Probes for Run 43 and Run 44

	Run 43 and Run 44	Run 43	Run 44
	Axial Position (z)	Radial Position (r)	Radial Position (r)
Probe #	(cm)	(cm)	(cm)
1 and 8	3 and 3	0 and 8.2	0 and 5.9
2 and 9	6 and 6	15.5 and 8.2	10.8 and 5.9
3 and 10	9 and 9	0 and 14	0 and 9.3
4 and 11	12 and 12	15.5 and 14	10.8 and 9.3
5 and 12	15 and 15	8.25 and 0	5.9 and 0
6 and 13	18 and 18	8.25 and 15.5	5.9 and 10.8
7 and 14	21 and 21	15.5 and 15.5	10.8 and 10.8

Using the temperature-concentration relationship, it was possible to construct a visualization of the concentration profiles within the beds at specified points in time. Shown in Figures 6 and 7 are the results for Run 43 and Run 44, respectively. The bed diameters are to scale. These plots were constructed by inferring the position of the concentration front based on the response of each of the 14 temperature probes. These profiles represent the position of the leading edge of the R-22 concentration front within the bed.

4. SUMMARY

The magnitude of the temperature swings within PSA system temperatures measured at a specific radial position during a cycle were converted to estimated concentrations using an empirically derived relationship between the R-11 vapor-phase concentration and the magnitude of the temperature changes. Using this approach, it was possible to construct a three-dimensional plot of vapor-phase concentrations with time and bed location (both axial and radial). Several important applications of this capability include: (1) identifying dispersion problems which can degrade separation efficiency; (2) monitoring long-term adsorbent degradation; and (3) providing critical temperature data to develop better mathematical models.

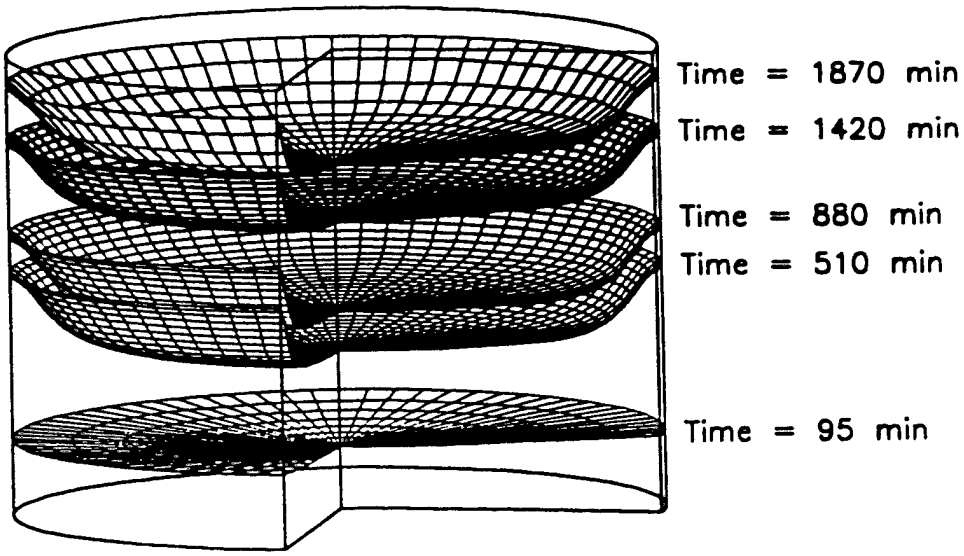

Figure 6. Shape of the R-22 Concentration Front at Selected Times for Run 43

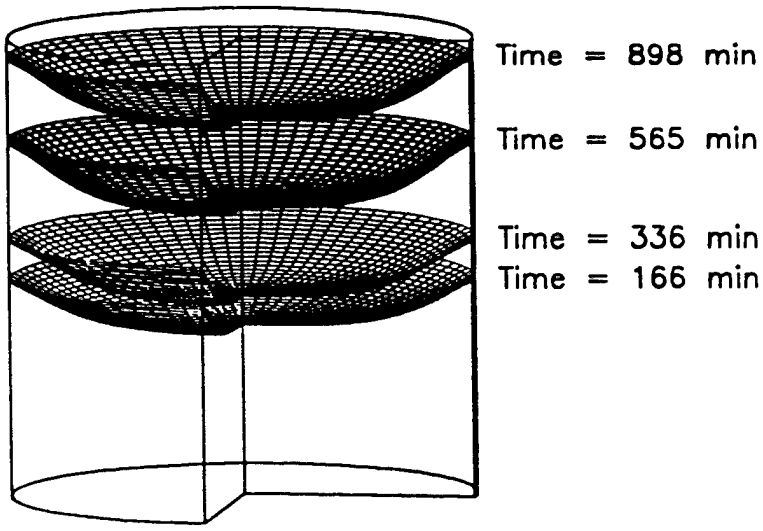

Figure 7. Shape of the R-22 Concentration Front at Selected Times for Run 44

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